

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2004-268152  
(43)Date of publication of application : 30.09.2004

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(51)Int.Cl. B24D 11/00  
B24D 3/00  
// B24B 19/00  
B24B 21/00

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## (54) POLISHING FILM

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a polishing film for the final finishing, its use, a polishing method using the polishing film, and a polishing product polished by the polishing film for working parts in high polishing surface quality without causing a scratch, a work level difference, and resin sticking on the polishing film, particularly, an optical fiber connector ferrule end surface, for working the electronic part and the optical part.

**SOLUTION:** In this polishing film, a polishing layer including an abrasive particle and a binder resin is arranged on a base material. The abrasive particle is an acid oxide particle, and surface treatment or wetting treatment is performed on the polishing particle by an amine compound.

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## LEGAL STATUS

[Date of request for examination] 01.03.2006

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]



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## CLAIMS

[Claim(s)]

[Claim 1]

The abrasive film which this abrasives particle is an acidic-oxide particle, and is characterized by this polish layer containing an amine compound in the abrasive film which prepared the polish layer containing an abrasives particle and binder resin on the base material.

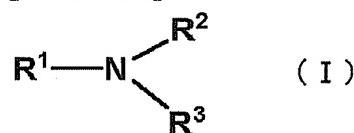
[Claim 2]

An abrasives particle is surface treatment or the abrasive film according to claim 1 by which humid processing is carried out by said amine compound.

[Claim 3]

The abrasive film according to claim 1 or 2 said whose amine compounds are 1 expressed with a structure expression (I) – a tertiary amine compound.

[Formula 1]

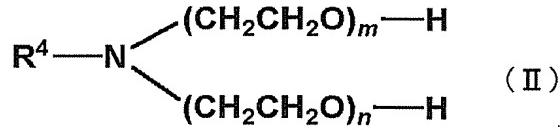


R1 shows the hydrocarbon group or permutation hydrocarbon group of the carbon atomic numbers 1–20 among a formula, and R2 and R3 show independently the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom or the carbon atomic numbers 1–20.

[Claim 4]

The abrasive film according to claim 1 or 2 said whose amine compound is the alkanolamine expressed with a structure expression (II), or a polyoxyethylene amine compound.

[Formula 2]



R4 shows the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom and the carbon atomic numbers 1–20 among a formula. m and n are the integers of 0–8, and are m+n=1–8.

[Claim 5]

The abrasive film of claim 1–4 which is at least one sort chosen from the group which an acidic oxide becomes from a silica, tin oxide, and a zinc oxide given in any 1 term.

[Claim 6]

The abrasive film of claim 1–5 which is a spherical granulation particle with a mean particle diameter of 1–10 micrometers which an abrasives particle becomes from a primary particle with a mean particle diameter of 20–30nm given in any 1 term.

[Claim 7]

The abrasive film according to claim 6 which is the granulation particle from which the spherical granulation particle carried out spray drying of the dispersion liquid of a primary particle, and was



obtained, or calcinates this at the temperature of 900 degrees C or less, and is obtained.  
[Claim 8]

The abrasive film of claim 1-7 whose acidic oxide is a silica given in any 1 term.

[Claim 9]

The polish approach characterized by using the abrasive film of any 1 term of claims 1-8.

[Claim 10]

The polish approach of the electronic parts or the optic characterized by using the abrasive film of any 1 term of claims 1-8.

[Claim 11]

Abrasive products ground using the abrasive film of any 1 term of claims 1-8.

[Claim 12]

Abrasive products according to claim 11 which are electronic parts or an optic.

[Claim 13]

Use of the abrasive film of claim 1-8 for polish of an optical connector ferrule end face given in any 1 term.

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[Translation done.]



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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

#### [0001]

##### [Field of the Invention]

This invention is an abrasive film for processing electronic parts, an optic, etc., and relates to the abrasive film suitable for using for a semi-conductor wafer, the ceramics, an optical lens, a magnetic-disk substrate, the magnetic head, the last finishing polish process of the color filter for liquid crystal, especially the last finishing polish process of an optical connector ferrule end face.

This invention relates to use of the polish approach which used the above-mentioned abrasive film, the abrasive products ground using this, and the above-mentioned abrasive film again.

#### [0002]

##### [Description of the Prior Art]

In recent years, the optical fiber used for the optical communication which is one of the information-transmission approaches is used through connection of connectors, in order to transmit a signal over long distances. Therefore, importance is attached to the dependability of a connection and improvement in the optical property called the insertion loss of light and reflection loss (return loss) of light has been a technical problem.

Since connection of connectors needs to contact a connector end face and needs to connect in the state of full adhesion by the suitable pressure, in recent years, what is called PC (Physical Contact) polish connector which carried out polish processing is used for the shape of the convex spherical surface in the connector end face which is the connection part of an optical fiber, and this edge surface part has the structure where of glass fiber was inserted into the cylinder called the ferrule made in zirconia ceramics.

#### [0003]

Usually, after it removes the adhesives which joined glass fiber to the ferrule using the abrasive film which used silicon carbide as an abrasives particle first, the polish process of this PC polish connector performs rough polish with a diamond abrasive film with a mean particle diameter of about 3 micrometers, subsequently gives inside polish with a diamond abrasive film with a mean particle diameter of about 1 micrometer, and, finally performs finishing polish.

however, a ferrule and glass fiber had a hardness difference between ingredients, and when the amount of polishes of the glass fiber which is elasticity becomes high, fiber drawing in (processing level difference) by superfluous polish (selection polish between different degree-of-hardness ingredients) of glass fiber occurs and connector end faces connect from zirconia ceramics, after the opening by the level difference was generated and reflective attenuation (the fall of return loss) of a lightwave signal arose according to the difference of the refractive index of glass fiber and an opening, there was a problem which acquires and says.

#### [0004]

In order to prevent selection polish of a different-species degree-of-hardness ingredient generally, it is known that what is necessary is just to use abrasives with the more high grinding force, for example, diamond abrasives etc. However, in processing by the high abrasives of the grinding force, although polish capacity improves, polish cracks, such as a minute scratch, occur in that machined surface, and this polish crack also becomes the cause of reducing return loss like a processing level difference.

Conventionally, in the last finishing polish of an optical connector ferrule end face, the abrasive

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film which distributed abrasives, such as a diamond, and an alumina, a silica, in suitable binder resin was used on base materials, such as mechanochemical polishing, polyester film, etc. using a polish slurry and abrasive cloth.

[0005]

For example, in the patent reference 1, the stiffness value of an abrasive film, surface roughness, and diamond particle diameter are controlled, the scratch on the front face of an optical fiber and generating of processing distortion are suppressed in it, using diamond abrasives as abrasives, and the diamond abrasive film with which return loss can obtain an optical connector 40dB or more is indicated.

Moreover, the abrasive film which formed the polish layer on the abrasive film base material with the coating liquid which distributed the silica particle whose mean particle diameter is 10–100mmicro in silicone resin liquid is indicated by the patent reference 2. Furthermore, in the patent reference 3 and the patent reference 4, the class of abrasives particle, mean particle diameter, the surface roughness of an abrasive film, transmission, the Hayes value, etc. are controlled, the scratch of an optical connector and generating of the processing level difference of processing distortion, a ferrule, and an optical fiber are suppressed in them, and the abrasive film with which the optical connector which raised return loss is obtained is indicated.

[Patent reference]

[Patent reference 1] JP,07-266240,A

[Patent reference 2] JP,08-336758,A

[Patent reference 3] JP,09-248771,A

[Patent reference 4] JP,10-071572,A

[0006]

In order to reduce generating of this processing level difference and polish crack as much as possible conventionally, formation of the spherical-surface configuration of a zirconia ferrule, \*\* of a connector end face – semifinishing polish were carried out with the abrasive film which used the about 1-micron diamond timely from the mean particle diameter of 6 microns as abrasives, and the polish process which used together the slurry which distributed the colloidal silica particle in the alkaline solution as last finishing polish, and abrasive cloth was used. However, as for management of a slurry, or its handling, it was complicated that there was the need of condensation of an abrasives particle occurring by change of the slurry concentration under polish processing, and washing a processing side after polish termination etc.

[0007]

Therefore, although processing using an abrasive film generally came to be used, it is mentioned that the binder resin of an abrasive film carries out transition adhesion at an optical connector ferrule end face, and produces an optical transmission error as a trouble in finishing polish processing which used the abrasive film. Since the glass fiber part contains silicon, such as quartz glass, when the silane system coupling agent and silicone resin containing silicon are used for binder resin, since binder resin peels from an abrasive film since polish processing is performed while the binder resin of an abrasive film contacts an optical connector ferrule end face directly, and adhesion in an optical connector ferrule end face takes place, this has a possibility of especially becoming easy to produce firm adhesion from both compatibility.

Homogeneity needs to distribute, if the floc of an abrasives particle exists, selection polish of a scratch or an elasticity ingredient will arise by polish processing, and it becomes impossible moreover, for an abrasives particle to attain the good polished surface grace by polish processing in the polish layer of an abrasive film.

[0008]

[Problem(s) to be Solved by the Invention]

The purpose of this invention is offering the abrasive film for processing electronic parts, an optic, etc.

Other purposes of this invention are offering the last finishing abrasive film into which the Koken facet quality's is processed, and its use, without producing a scratch, a processing level difference, and resin adhesion in an optical connector ferrule end face.

The purpose of further others of this invention is offering the polish approach which used the above-mentioned abrasive film.

The purpose of further others of this invention is offering the abrasive products ground with the above-mentioned abrasive film.



[0009]

[Means for Solving the Problem]

This invention offers the abrasive film shown below, the polish approach, and abrasive products.

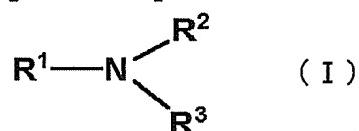
1. Abrasive film which this abrasives particle is acidic-oxide particle, and is characterized by this polish layer containing amine compound in abrasive film which prepared polish layer containing abrasives particle and binder resin on base material.

2. An abrasives particle is the abrasive film of surface treatment or one above-mentioned publication by which humid processing is carried out by the amine compound.

3. Abrasive film of the above 1 said whose amine compounds are 1 expressed with structure expression (I) – tertiary amine compound, or two publications.

[0010]

[Formula 3]



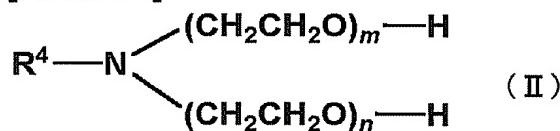
[0011]

R1 shows the hydrocarbon group or permutation hydrocarbon group of the carbon atomic numbers 1–20 among a formula, and R2 and R3 show independently the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom or the carbon atomic numbers 1–20.

4. Abrasive film of the above 1–3 said whose amine compounds are alkanolamine expressed with structure expression (II), or polyoxyethylene amine compound given in any 1 term.

[0012]

[Formula 4]



[0013]

R4 shows the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom and the carbon atomic numbers 1–20 among a formula. m and n are the integers of 0–8, and are m+n=1–8.

5. Abrasive film of the above 1–4 which is at least one sort chosen from group which acidic oxide becomes from silica, tin oxide, and zinc oxide given in any 1 term.

6. Abrasive film of the above 1–5 which is spherical granulation particle with a mean particle diameter of 1–10 micrometers which abrasives particle becomes from primary particle with a mean particle diameter of 20–30nm given in any 1 term.

7. Abrasive film of six above-mentioned publication which is granulation particle from which spherical granulation particle carried out spray drying of dispersion liquid of primary particle, and was obtained, or calcinates this at temperature of 900 degrees C or less, and is obtained.

8. Abrasive film of the above 1–7 whose acidic oxide is silica given in any 1 term.

9. Polish approach characterized by using abrasive film of any 1 term of the above 1–8.

10. The polish approach of of the electronic parts or the optic characterized by using the abrasive film of any 1 term of the above 1–8.

11. Abrasive products ground using the abrasive film of any 1 term of the above 1–8.

12. Abrasive products of 11 above-mentioned publication which is electronic parts or an optic.

13. Use of the abrasive film of the above 1–8 for polish of an optical connector ferrule end face given in any 1 term.

[0014]

[Function]

The polish layer of the abrasive film of this invention is formed on a base material by applying the coating liquid which contains the acidic oxide which is an abrasives particle, for example, a silica, an amine compound, and binder resin, and it comes to distribute. Since the front face of



the silica distributed by the solvent of coating liquid is charged in negative, the amine compound which has positive charge sticks to the silica front face which has a negative charge with electric attraction. For this reason, it can prevent a silica contacting an optical fiber end face directly in polish processing. Moreover, since it sticks to an amine compound alternatively and it forms a protective layer in the glass fiber front face which consists of quartz glass, the polish rate of a glass fiber part falls, and the polish impact on the front face of glass fiber which is an elasticity ingredient is reduced. It is thought that polish processing is carried out without producing a lubrication action, preventing a processing level difference as a result, and producing a scratch and processing distortion, when an amine compound furthermore intervenes.

#### [0015]

On the other hand, a zirconia ferrule is an alkaline oxide, when inorganic oxides of a different kind, such as an acidic oxide and an alkaline oxide, contact, the addition reaction accompanied by electronic transfer arises in the interface, and the layer of a multiple oxide is formed in a zirconia front face. It is thought that the polish removal rate of a zirconia rises and the layer of this multiple oxide prevents generating of the processing level difference between the dissimilar materials in an optical connector ferrule end face since a degree of hardness falls rather than the original zirconia. Furthermore, since an amine compound shows alkalinity, the front face of the silica which is an acidic oxide is activated by the chemical operation. For this reason, since the interface reaction nature of the silica which is an abrasives particle, and the zirconia which is a ground object becomes high and a conjugated compound becomes is easy to be formed, the polish removal rate of a zirconia becomes higher and it is thought that generating of the processing level difference between dissimilar materials is prevented more effectively.

For this reason, it is thought that it becomes possible to acquire the high-definition polished surface which reduced the processing level difference, without a scratch and processing distortion occurring in a finishing polish process by using the abrasive film of this invention.

#### [0016]

##### [Embodiment of the Invention]

Hereafter, the gestalt of operation of this invention is explained to a detail.

As a base material used for this invention, although plastic film, such as polyester, such as polyethylene terephthalate and polybutylene terephthalate, polyimide, a polycarbonate, polyethylene, polypropylene, polystyrene, a polyvinyl chloride, a polyvinylidene chloride, a polyacrylonitrile, acrylic resin, and two or more sorts of such mixture, can be used, for example, polyester film is desirable as a base material excellent in the mechanical strength and dimensional stability at the time of the thermal resistance at the time of abrasive film production, or polish. Moreover, films which carried out surface treatment, such as corona discharge and plasma discharge, can also be used.

25–150 micrometers of thickness of a base material are 50–100 micrometers more preferably that what is necessary is just the range which has the mechanical strength and elasticity which follows in footsteps of a polished surface effectively of extent which is not damaged to the pressurization at the time of polish processing etc.

#### [0017]

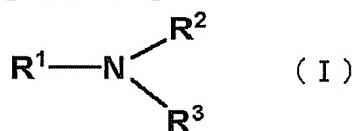
The abrasives particles used for this invention are an acidic oxide, especially an acid metallic oxide, for example, a silica, tin oxide ( $\text{SnO}_2$ ), zinc oxides, two or more sorts of such mixture, etc. are mentioned. To polish of an optical connector ferrule end face, a silica is especially desirable.

#### [0018]

Especially the desirable amine compounds used for this invention are 1 expressed with the following structure expression (I) – a tertiary amine compound.

#### [0019]

##### [Formula 5]



#### [0020]

R1 shows the hydrocarbon group or permutation hydrocarbon group of the carbon atomic



numbers 1–20 among a formula, and R2 and R3 show independently the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom or the carbon atomic numbers 1–20. As a hydrocarbon group, the saturation of a straight chain or branching or the aliphatic hydrocarbon radical of partial saturation, an aliphatic–cyclic–hydrocarbon radical, and an aromatic hydrocarbon radical are mentioned. As a substituent of a permutation hydrocarbon group, the alkoxy group of the carbon atomic numbers 1–20, an alkenyloxy radical, etc. are mentioned.

[0021]

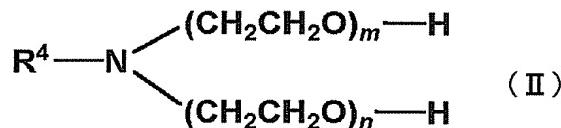
As an example of a 1 – tertiary amine compound expressed with a structure expression (I) Triethylamine, propylamine, a butylamine, dibutyl amine, Tributylamine, n amylamine, s–amyl amine, t–amyl amine, An iso–amyl amine, n–hexylamine, a heptyl amine, n–octyl amine, s–octyl amine, t–octyl amine, trioctylamine, 2–ethylhexylamine, A DESHIRU amine, a dodecyl amine, tetradecylamine, a hexadecyl amine, A stearyl amine, an oleyl amine, dodecyl dimethylamine, tetradecyl dimethylamine, Hexadecyl dimethylamine, octadecyl dimethylamine, 3–ethoxypropylamine, 3–(2–ethylhexyloxy) propylamine, o–, m–, or p–butyl aniline, o–, m– or p–hexyl aniline, o–, m– or p–octyl aniline, o–, m– or p–dodecyl aniline, o–, m– or p–tetradecyl aniline, o–, m– or p–ethoxy anilines, two or more sorts of such mixture, etc. are mentioned. The above-mentioned alkyl group, an alkenyl radical, an alkoxy group, and an alkenyloxy radical shall also mean all position isomers and optical isomers. Moreover, this invention is not limited to these.

[0022]

Moreover, as other desirable amine compounds used for this invention, the monochrome expressed with the following structure expression (II), JI, a thoria RUKANORU amine, or a polyoxyethylene amine compound is mentioned.

[0023]

[Formula 6]



[0024]

R4 shows the hydrocarbon group or permutation hydrocarbon group of a hydrogen atom and the carbon atomic numbers 1–20 among a formula. m and n are the integers of 0–8, and are m+n=1–8. As a hydrocarbon group, the saturation of a straight chain or branching or the aliphatic hydrocarbon radical of partial saturation, an aliphatic–cyclic–hydrocarbon radical, and an aromatic hydrocarbon radical are mentioned. As a substituent of a permutation hydrocarbon group, the alkoxy group of the carbon atomic numbers 1–20, an alkenyloxy radical, etc. are mentioned.

[0025]

As an example of the monochrome expressed with a structure expression (II), JI, a thoria RUKANORU amine, or a polyoxyethylene amine compound, a hydroxy ethylene dodecyl amine, a polyoxyethylene dodecyl amine, a polyoxyethylene octadecyl amine, a polyoxyethylene oleyl amine, monoethanolamine, diethanolamine, triethanolamine, etc. are mentioned. This invention is not limited to these.

[0026]

It is possible independent or for the abrasive film which added two or more kinds at a rate of arbitration to attain the Koken facet quality for these amine compounds.

The addition concentration of these amine compounds used for this invention is the range of the 0.5 – 20.0 mass section to the abrasives particle 100 mass section, and is the 1.0 – 10.0 mass section preferably. If there are few additions of these additives than the 0.5 mass section, being [ which is made into the purpose / of effectiveness ] discovered will become inadequate, and there is a possibility that a scratch may arise. Moreover, even if it adds exceeding the 20.0 mass sections, the further improvement in effectiveness is not found.

[0027]

What is necessary is just to add an amine compound suitably in the process of the arbitration of the coating liquid manufacture for polish layers in the manufacture process of the abrasive film of this invention. For example, what is necessary is to add the initial complement of an abrasives



particle and an amine compound in the resin liquid which dissolved binder resin with the solvent, and just to carry out mixed distribution. Moreover, weighing capacity of an abrasives particle and the amine compound is carried out, the solvent of optimum dose is added, stirring mixing is carried out, the resin liquid which dissolved binder resin in this with the solvent may be added, and mixed distribution may be carried out. this -- an abrasives particle -- an amine compound -- surface treatment -- or humid processing can be carried out.

[0028]

Moreover, as an abrasives particle of this invention, a spherical granulation particle with a mean particle diameter of 1–10 micrometers corned and produced by spray drying can also use the dispersion liquid of a primary particle with a particle diameter of 20–30nm. This spherical granulation particle can control the pore volume and the particle diameter of a granulation particle by the particle concentration of the dispersion liquid of a primary particle, and the spraying approach to arbitration, and can adjust with drying temperature, the bonding strength, i.e., the particle degree of hardness, of primary particles.

The mean particle diameter of the spherical granulation particle used for this invention is 0.5–30.0 micrometers preferably, and is 1.0–10.0 micrometers more preferably. The particle diameter of a granulation particle influences the amount of polish removal, and in less than 0.5 micrometers, when it has the inclination for the polish force to become low and it exceeds 30.0 micrometers, it has the inclination for the processing level difference produced with the difference of grinding quantity between the ingredients of a different degree-of-hardness ingredient to become large under the effect of the surface waviness component of a film.

[0029]

Moreover, the pore volume of the granulation particle used for this invention has desirable 0.1 – 1.0 cc/g. In less than 0.1 cc/g, since a primary particle will be in a dense state of aggregation, the degree of hardness of a granulation particle is large and not a primary particle but a granulation particle acts as one abrasives particles as \*\* and a result, there is an inclination for the processing level difference produced with the difference of grinding quantity between the ingredients of a different degree-of-hardness ingredient to become large. If 1.0 cc/g is exceeded, since a primary particle will be in a \*\*\*\* state of aggregation and the degree of hardness of a granulation particle will become small, a granulation particle will be crushed by pressurization under polish processing, it will be destroyed, and there is an inclination normal polish becomes impossible.

[0030]

Although the granulation particle used for this invention may be used with un–calcinating, preferably, it is 420 degrees C or less, and 900 degrees C or less of things calcinated under ambient atmospheres, such as oxygen, air, and inert gas, can also be used for 1 to 5 hours. If burning temperature exceeds 900 degrees C, since it will become large, the bonding strength, i.e., the particle degree of hardness, of primary particles, and not a primary particle but a granulation particle will act as one abrasives particle as a result, there is an inclination for the processing level difference produced with the difference of grinding quantity between the ingredients of a different degree-of-hardness ingredient to become large. moreover, the thing which the silanol group of a silica particle front face will carry out dehydration condensation, and will disappear if it exceeds 420 degrees C -- as (bibliography -- Journal of Colloid Science 13 [ ] -- 67–85 [1958] etc. --) -- there is an inclination for the chemical-polishing operation by glass and the hydrophilic silanol group at the time of polish processing of a zirconia to no longer be acquired.

[0031]

Since such a granulation particle has the comparatively large abrasives particle and it can fix on a base material, without burying an abrasives particle in binder resin like the abrasive film which makes binder resin come to distribute the conventional particle At the time of polish processing, operating stress is large in the contact section with a ground object, the high polish force is produced, on the other hand, it is cracked by the primary particle in the contact section by this operating stress, wear discharge is carried out, and polish processing is carried out in the state of an always uniform primary particle. Therefore, it is thought that it excels in the capacity to attain the Koken facet quality. Moreover, since a ground object front face and binder resin do not contact directly, resin adhesion can be prevented and it is more effective. Since it furthermore craters in a polish layer and a part exists, generating of the scratch by the discard which does not act on polish is controlled by discharging wear waste and polish waste of an



abrasives particle efficiently from a polished surface.

Since the spherical \*\* style particle of the abrasive film of this invention is the floc of a primary particle, when the processing pressure force of joining an abrasive film from a ground object is eased by the crack to a primary particle, it is thought that processing distortion, i.e., generating of a damaged layer, is reduced.

[0032]

Thermosetting resin and thermoplastics are mentioned as binder resin used for this invention. As thermosetting resin, polyester resin, polyester polyol resin, polyurethane resin, polyester polyurethane resin, an epoxy resin, phenoxy resin, phenol resin, a urea-resin, etc. are mentioned. As thermoplastics, the urethane elastomer which is acrylic resin, nitrocellulose system resin, nitrocellulose-acrylic resin, denaturation acrylic resin, an alkyd resin, polyolefine system resin, polyester system resin, and rubber system resin, nitrile rubber, ethylene vinyl acetate rubber, fluororubber system resin, other water soluble resin, emulsion resin, etc. are mentioned.

Chemical modification of these resin may be carried out by the need by functional groups (a hydroxyl group, the amino group, carboxyl group, etc.). Binder resin is used at a rate of independent or two or more kind arbitration in these resin.

In this invention, it is appropriate for binder resin to carry out 30-70 mass section extent use to the total quantity 100 mass section of an abrasives particle and an amine compound.

[0033]

A curing agent is used for the coating liquid for forming the polish layer of this invention if needed. The poly isocyanate is mentioned as an example of a curing agent. For example, hexamethylene di-isocyanate, tolylene diisocyanate, xylylene diisocyanate, isophorone diisocyanate, naphthylene diisocyanate, etc. are mentioned. These curing agents are used at a rate of independent or two or more kind arbitration.

[0034]

Although especially the solvent used for the coating liquid for forming the polish layer of this invention is not limited, an acetone, a methyl ethyl ketone, methyl isobutyl ketone, a cyclohexanone, an isophorone, ethyl acetate, butyl acetate, a tetrahydrofuran, toluene, a xylene, ethanol, isopropanol, etc. are mentioned, for example. These organic solvents can dissolve binder resin and a curing agent in homogeneity, and further, an abrasives particle is distributed by stability, and after spreading, they are used at a rate of independent or two or more kind arbitration so that desiccation removal may be carried out completely.

Moreover, other surfactants, coupling agents, etc. can be used for the coating liquid for forming the polish layer of this invention if needed.

[0035]

In the resin liquid which dissolved binder resin with the solvent, in the initial complement of an abrasives particle and an amine compound, it mixes, and it is dispersers, such as a sand mill, and the abrasive film of this invention is manufactured weighing capacity and by distributing to homogeneity in resin liquid and applying an abrasives particle on a base material. The rate of the solid content in coating liquid is 30 to 70 mass % preferably. As a spreading means to a base material top, a wire bar coating machine, a gravure coating machine, a reverse coating machine, a knife coating machine, a nozzle, a die coating machine, etc. can be used, for example. A polish layer fixes to a base material by applying coating liquid on a base material, so that it may become about two 5 - 20 g/m with dry mass, and drying or hardening processing at 40-120 degrees C with such a spreading means, for 0.5 to 72 hours.

[0036]

[Example]

Hereafter, an example explains this invention still more concretely.

An example 1, the examples 1 and 2 of a comparison

Polyester polyurethane system resin UR1350 (Toyobo [ Co., Ltd. ] make: solid content 33 mass %) (35 mass sections) was dissolved in the methyl ethyl ketone (44 mass sections) as binder resin, and resin liquid was produced. After having added fumed silica (grinding silica) (FQ3000: Fujimi Make, mean particle diameter of 3-4 micrometers) (20 mass sections) as an abrasives particle, adding the 1st class amine (compound name POE(7) dodecyl amine) (NAIMIN L-207: Nippon Oil & Fats Co., Ltd. make) (1 mass section) to this as an additive and distributing to homogeneity by the sand mill, filtering of 75 micrometers of filtration accuracies was given, and coating liquid was produced.



## [0037]

The \*\* which does not add the 1st class amine as an additive in an example 1 as an example 1 of a comparison, What made the amount of binder resin 36 mass sections, As the abrasives particle (ORGANO silica sol MEK-ST (30 mass %)) (80 mass sections) (solvent methyl ethyl ketone) and binder resin with which average grain size consists of a particle silica which is about 50nm as an example 2 of a comparison Mixed stirring processing of the silicone resin (KR-9218: Shin-Etsu Chemical Co., Ltd. make) (20 mass sections) was carried out, and coating liquid was produced.

The produced coating liquid was applied by the knife coating machine on the polyethylene terephthalate film (SG-2: by Teijin, Ltd.) with a thickness of 75 micrometers which performed easily-adhesive processing (what applied about 0.1 micrometers of polyester system resin). The drying-furnace temperature of a coating machine was set as 120 degrees C, and carried out room temperature neglect after spreading for 24 hours or more. Spreading thickness after desiccation was set to 5 micrometers.

## [0038]

Examples 2-8, the examples 3-8 of a comparison

Next, the example using the spherical granulation particle as an abrasives particle is described. Resin liquid was produced as an organic solvent, using polyester polyurethane system resin UR1350 (Toyobo [ Co., Ltd. ] make: 33 mass %) (35 mass sections) as a methyl ethyl ketone (44 mass sections) and binder resin. After carrying out mixed stirring of the additive (1 mass section) shown with the silica granulation abrasives particle P500 (catalyst formation industrial incorporated company make) (20 mass sections) in Table 1 as an abrasives particle at this and distributing to homogeneity by the sand mill, filtering of 75 micrometers of filtration accuracies was given, and coating liquid was produced.

## [0039]

Examples 2 and 3 are the 1st class amines, and examples 4 and 5 are tertiary amine, an example 6 - 8 alkylamine ethylene oxide derivative. The example 3 of a comparison has not added the additive. Moreover, although all examples are amine compounds (all are the Nippon Oil & Fats Co., Ltd. make), the examples 4-6 of a comparison are acid compounds (made in Nikko Chemicals, Inc.), and the examples 7 and 8 of a comparison are neutral compounds (Kao Corp. make).

Coating liquid was applied by the knife coating machine on the polyethylene terephthalate film (SG-2: Teijin, Ltd. make) with a thickness of 75 micrometers which performed easily-adhesive processing. The drying-furnace temperature of a coating machine was set as 120 degrees C, and carried out room temperature neglect after spreading for 24 hours or more. Spreading thickness after desiccation was set to 5 micrometers.

## [0040]

[Table 1]



	化合物名	商品名
実施例2	ドデシルアミン	アミンBB
実施例3	オレイルアミン	アミンOB
実施例4	テトラデシルジメチルアミン	3級アミンMB
実施例5	オクタデシルジメチルアミン	3級アミンABT
実施例6	ヒドロキシエチルドデシルアミン	ナイミーンL-201
実施例7	POEドデシルアミン	ナイミーンL-202
実施例8	POEドデシルアミン	ナイミーンL-207
比較例3	—	—
比較例4	POE(7)トリデシルエーテル酢酸	ECT-7
比較例5	ジPOE(2)アルキルエーテルリン酸	DDP-2
比較例6	ジPOE(6)アルキルエーテルリン酸	DDP-6
比較例7	POEノニルフェニルエーテル	NP-2
比較例8	POEノニルフェニルエーテル	NP-10

## [0041]

About the abrasive film of an example and the example of a comparison, finishing polish of an optical connector ferrule end face was performed according to the usual polish process. After the polish process performed adhesives removal with the abrasive film which used silicon carbide first, it gave rough polish with the diamond abrasive film with a grain size of about 3 micrometers, subsequently gave inside polish with the diamond abrasive film with a grain size of about 1 micrometer, and, finally performed finishing polish with the abrasive film of an example and the example of a comparison.

Evaluation of a polished surface evaluated a scratch and resin adhesion by the differential interference microscope (Axio Plan made from curl TSUAISU, observation scale-factor x510.2). The processing level difference measured the level difference between a zirconia ferrule and an optical fiber with the atomic force microscope (AFM, Explorer2000 made from Topo Metrix, and scanning area 100micrometer). Return loss is Optical made from EXPO. Test System IQ-2D3 is used and it is JIS. C5961 It carried out by the optical branching device method by the optical connector examining method.

The value of return loss,

$$RL=10\log_{10}(P1/P0)[dB]$$

Be alike is computed. Here, the optical power which P0 inputted, and P1 show the reflected optical power, respectively. If the value of return loss assumes that it is -10dB and 500-microwatt light will carry out incidence to an optical fiber end face, 50-microwatt light will reflect toward the light source. That is, 10% of energy will lose. A result is shown in Table 2.

## [0042]

[Table 2]



	スクラッチ	加工段差	樹脂付着	反射減衰量
実施例1	ややあり	-29 nm	なし	59 dB
比較例2	なし	+10 nm	あり	57 dB
比較例3	あり	-81 nm	なし	42 dB
実施例2	なし	-2 nm	なし	62 dB
実施例3	なし	-5 nm	なし	60 dB
実施例4	なし	-20 nm	なし	60 dB
実施例5	なし	-18 nm	なし	60 dB
実施例6	なし	-5 nm	なし	66 dB
実施例7	なし	-8 nm	なし	63 dB
実施例8	なし	-13 nm	なし	62 dB
比較例3	あり	-110 nm	なし	58 dB
比較例4	あり	-153 nm	なし	52 dB
比較例5	あり	-162 nm	なし	55 dB
比較例6	あり	-109 nm	なし	53 dB
比較例7	あり	-89 nm	なし	55 dB
比較例8	あり	-81 nm	なし	53 dB

## [0043]

As a result of evaluating, in the example 1 of a comparison which did not add an amine compound to the example 1, the cracks of glass fiber and a zirconia ferrule also occurred frequently, and return loss was also low. Resin adhesion generated the example 2 of a comparison using the abrasives particle and silicone resin with which average grain size consists of a particle silica which is about 50nm. In the abrasive film which added the amine compound of an example 1, although some crack remained in glass fiber, since the zirconia ferrule did not have a crack, either and return loss also became higher than the example 1 of a comparison, it was checked that it is possible to raise polish quality and an optical property according to the effectiveness of an amine compound.

## [0044]

The abrasive film of examples 2–8 using the spherical granulation particle as an abrasives particle did not have the scratch of a glass fiber part, a processing level difference is also 20nm or less, and resin adhesion was not accepted, either. Moreover, all return loss is 60dB or more, and was able to carry out precision polish with very little attenuation of a lightwave signal. On the other hand, although there is no resin adhesion, the scratch has generated the abrasive film of the examples 3–8 of a comparison, and a processing level difference is large and return loss is also about 55dB owing to it.

## [0045]



**[Effect of the Invention]**

If the abrasive film of this invention is used for the last finishing polish of a fiber-optic connector ferrule end face as mentioned above, a high-definition polished surface can be acquired without producing a scratch, a processing level difference, and resin adhesion in an optical connector ferrule end face.

Moreover, in the abrasive film using the spherical granulation particle as an abrasives particle, a processing level difference is 20nm or less, return loss is 60dB or more, and attenuation of a lightwave signal can carry out very little precision polish.

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[Translation done.]



(19) 日本国特許庁(JP)

## (12) 公開特許公報(A)

(11) 特許出願公開番号

特開2004-268152  
(P2004-268152A)

(43) 公開日 平成16年9月30日(2004.9.30)

(51) Int.Cl.<sup>7</sup>  
**B24D 11/00**  
**B24D 3/00**  
// **B24B 19/00**  
**B24B 21/00**

F I  
B 24 D 11/00      B  
B 24 D 3/00      320 A  
B 24 D 3/00      330 A  
B 24 D 3/00      330 D  
B 24 D 3/00      350

テーマコード(参考)

3C049  
3C058  
3C063

審査請求 未請求 請求項の数 13 O L (全 13 頁) 最終頁に続く

(21) 出願番号  
(22) 出願日

特願2003-58202 (P2003-58202)  
平成15年3月5日 (2003.3.5)

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(54) 【発明の名称】研磨フィルム

(57) 【要約】 (修正有)

【課題】電子部品、光学部品等を加工するための研磨フィルム、特に光ファイバコネクタフェルール端面にスクラッチや加工段差、樹脂付着を生じることなく高研磨面品質に加工する最終仕上げ用研磨フィルム、その使用、研磨フィルムを用いた研磨方法、及び研磨フィルムにより研磨された研磨製品を提供する。

【解決手段】基材上に、研磨材粒子とバインダ樹脂を含む研磨層を設けた研磨フィルムにおいて、研磨材粒子が酸性酸化物粒子であり、研磨粒子がアミン化合物により表面処理または湿潤処理されている。

【選択図】なし

【請求項13】

電子部品又汎光学部品で電子部品の研磨1用部品の研磨製品。

【請求項12】

請求項1～8のうち実机加工用機器で工具を用いた研磨工具の研磨製品。

【請求項11】

部品の研磨方法。

請求項1～8のうち実机加工用機器で工具を用いた特徴又は電子部品又汎光学

【請求項10】

請求項1～8のうち実机加工用機器で工具を用いた特徴又は研磨方法。

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【請求項9】

難燃性化物が工具で工具を請求項1～7のうち実机加工用部品の研磨で工具。

0.0°C以下の温度下で成る工具を請求項6用部品の研磨で工具。

【請求項7】

球状過渡粒子工具、一次粒子の分散液を噴霧乾燥して得た過渡粒子工具又は、工具を9

研磨材粒子工具、平均粒子径20～30nmの一次粒子工具平均粒子径1～10μmの

【請求項6】

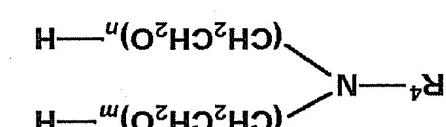
難燃性化物が、工具、難燃性化物又は難燃性化物を含む工具の少なからぬ量で工具

を請求項1～4のうち実机加工用部品の研磨で工具。

【請求項5】

及2n+1(0～8の整数)工具、m+n=1～8工具。

式中、R<sup>1</sup>汎水素原子、炭素原子数1～20の炭化水素基又は置換炭化水素基表示し。m



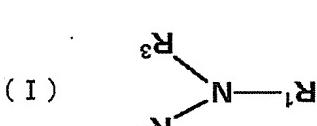
【化2】

2丁目～化合物工具を請求項1又は2用部品の研磨で工具。

前記丁目～化合物工具(II)工具又は工具の工具として又は工具として工具を1

【請求項4】

独立した、水素原子又は炭素原子数1～20の炭化水素基又は置換炭化水素基表示し。



【化1】

前記丁目～化合物工具(II)工具又は工具の工具を請求項1又は2

【請求項3】

工具として。

研磨材粒子工具前記丁目～化合物工具の表面処理又は置換処理工具の工具を研磨で工具。

【請求項2】

工具として。

基材上に、研磨材粒子工具又は研磨工具を被付ける工具の工具を有する工具又は工具

【請求項1】

【特許請求の範囲】

磨フィルムの使用。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】

本発明は、電子部品、光学部品等を加工するための研磨フィルムであり、半導体ウエハ、セラミックス、光学レンズ、磁気ディスク基板、磁気ヘッド、液晶用カラーフィルターの最終仕上げ研磨工程、特に光ファイバコネクタフェルール端面の最終仕上げ研磨工程に用いるのに適した研磨フィルムに関するものである。

本発明はまた、上記研磨フィルムを用いた研磨方法、これを用いて研磨された研磨製品、及び上記研磨フィルムの使用に関する。 10

【0002】

【従来技術の説明】

近年、情報伝送方法の一つである光通信に使用される光ファイバは、信号を長距離伝送するためにコネクタ同士の接続を介して使用されている。そのため接続部の信頼性が重要視され、光の挿入損失および光の反射損失（リターンロス）と云った光学的特性の向上が課題になっている。

コネクタ同士の接続は適当な圧力でコネクタ端面を接触させ、完全密着状態で接続させる必要があるため、近年では光ファイバの接続部分であるコネクタ端面を凸球面状に研磨加工したPC（Physical Contact）研磨コネクタと呼ばれるものが使用され、この端面部はジルコニアセラミックスで出来たフェルールと呼ばれる筒の中にガラスファイバが挿入された構造となっている。 20

【0003】

通常、このPC研磨コネクタの研磨工程は最初に炭化ケイ素を研磨材粒子として用いた研磨フィルムを用いてフェルールとガラスファイバを接合した接着剤の除去を行った後、平均粒子径 $3\text{ }\mu\text{m}$ 程度のダイヤモンド研磨フィルムで粗研磨を行い、次いで平均粒子径 $1\text{ }\mu\text{m}$ 程度のダイヤモンド研磨フィルムで中研磨を施し、最後に仕上げ研磨を行う。

しかしながらフェルールとガラスファイバとは材料間に硬度差があり、ジルコニアセラミックスより軟質であるガラスファイバの研磨量が高くなり、ガラスファイバの過剰研磨（異硬度材料間の選択研磨）によるファイバ引き込み（加工段差）が発生し、コネクタ端面同士を接続した時に段差による空隙が生じ、ガラスファイバと空隙との屈折率の差によつ 30 光信号の反射減衰（反射減衰量の低下）が生じてしまうという問題があった。

【0004】

一般に異種硬度材料の選択研磨を防止するためには、より研削力の高い研磨材、例えばダイヤモンド研磨材などを用いればよいことが知られている。しかし、研削力の高い研磨材による加工では、研磨能力は向上するものの、その仕上げ面には微小なスクラッチなどの研磨キズが発生し、この研磨キズも加工段差と同様に反射減衰量を低下させる原因となる。

従来、光ファイバコネクタフェルール端面の最終仕上げ研磨においては、研磨スラリーと研磨布を用いたメカノケミカル研磨やポリエステルフィルムなどの基材上にダイヤモンドやアルミナ、シリカなどの研磨材を適当なバインダ樹脂中に分散した研磨フィルムなどが 40 用いられていた。

【0005】

例えば、特許文献1には、研磨材としてダイヤモンド研磨材を用い、研磨フィルムのスティフネス値、表面粗さ、ダイヤモンド粒子径を制御して、光ファイバ表面のスクラッチや加工歪の発生を抑え、反射減衰量が40dB以上の光ファイバコネクタを得ることができるダイヤモンド研磨フィルムが開示されている。

また特許文献2には、研磨フィルム基材上に平均粒径が $10\sim100\text{ }\mu\text{m}$ のシリカ粒子をシリコーン樹脂液中に分散させた塗工液により研磨層を形成した研磨フィルムが開示されている。さらに特許文献3、特許文献4には、研磨材粒子の種類や平均粒子径、研磨フィルムの表面粗さ、透過率、ヘイズ値などを制御し、光ファイバコネクタのスクラッチや加 50

[0100]

<sup>2</sup> 諸種の機器にて云々。

3. 前記アミノ化合物の構造式(I)又表記丸1~3般アミノ化合物表記上記1又以  
テル。

22. 訓繕材料顯示加了三乙酸合物之後土壤表面處理及試驗調查處理之效果比之前上肥1倍較明顯。

11. 基材上に、研磨材粒子は $\mu\text{m}$ 程度の大きさで、表面粗さを $0.1\text{ }\mu\text{m}$ 以下に保つ。

本證明狀、以下証言有誤者二十八人、所據方法及証據繩品充據供詞之由以正為務。

【解題】本題是考究「先秦的爭戰」。

[ 6 0 0 0 ]

本證明的目的是證明上記所舉之幾何方法能提供正確的結果。

本說明書的目的地，光子干涉儀之主要構造及工作原理，並說明各項操作方法及各項之使用要訣。

【說明文解説】本圖是為了說明目的物、電子部品、光學部品等在加工方面的困難，以及它們所提供的工

【器物分解法之初步認識】

[ 8 0 0 0 ]

首先，研磨刀具上的研磨颗粒尺寸必须均匀一致，以满足加工过程中对研磨颗粒尺寸的要求。其次，研磨颗粒的硬度应适中，过大或过小都会影响研磨效果。

の光の研磨面で、工具の刃先を用いて工具の刃先を研磨する。研磨工具は、研磨面に光反射鏡付着工具と研磨面に研磨粉付着工具の二種類である。工具の刃先は、工具の刃先を研磨する工具の刃先と同一の形状である。

[ 2 0 0 0 ]

• 云の取引機会を増加する。

近年来，乙的加工精度与研磨工具的精度密切相关，研磨材料乙的平均粒子径6~10μm，乙的研磨效率与研磨工具的精度密切相关。乙的研磨效率与研磨工具的精度密切相关。乙的研磨效率与研磨工具的精度密切相关。

19000

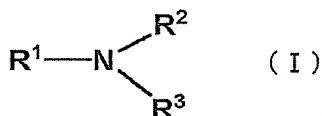
〔特許文献4〕特開平10-071572号公报

【特許文献3】特開平09-248771号公报

【精英文庫2】特輯平08-336758號公報

【轉譯文獻】轉譯平 07 - 266240 號公報

工业光与激光加工设备的装配、反斜键塞量检测上这些光门与接口未必获得充分利用而浪费了宝贵的集成化机会。



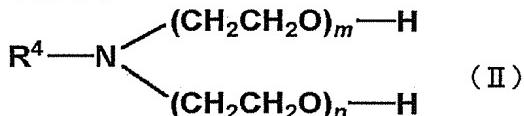
【 0 0 1 1 】

式中、 $\text{R}^1$ は炭素原子数1～20の炭化水素基又は置換炭化水素基を示し、 $\text{R}^2$ 、 $\text{R}^3$ は独立して、水素原子又は炭素原子数1～20の炭化水素基又は置換炭化水素基を示す。

4. 前記アミン化合物が構造式(I I)で表されるアルカノールアミン又はポリオキシエチレンアミン化合物である上記1～3のいずれか1項記載の研磨フィルム。

【 0 0 1 2 】

【化4】



【 0 0 1 3 】

式中、 $\text{R}^4$ は水素原子、炭素原子数1～20の炭化水素基又は置換炭化水素基を示す。 $m$ 及び $n$ は0～8の整数であり、 $m+n=1 \sim 8$ である。

5. 酸性酸化物が、シリカ、酸化錫及び酸化亜鉛からなる群から選ばれる少なくとも1種である上記1～4のいずれか1項記載の研磨フィルム。

6. 研磨材粒子が、平均粒子径20～30nmの一次粒子からなる平均粒子径1～10μmの球状造粒粒子である上記1～5のいずれか1項記載の研磨フィルム。

7. 球状造粒粒子が、一次粒子の分散液を噴霧乾燥して得られた造粒粒子であるか、これを900℃以下の温度で焼成して得られたものである上記6記載の研磨フィルム。

8. 酸性酸化物がシリカである上記1～7のいずれか1項記載の研磨フィルム。

9. 上記1～8のいずれか1項の研磨フィルムを用いることを特徴とする研磨方法。

10. 上記1～8のいずれか1項の研磨フィルムを用いることを特徴とする電子部品又は光学部品の研磨方法。

11. 上記1～8のいずれか1項の研磨フィルムを用いて研磨された研磨製品。

12. 電子部品又は光学部品である上記11記載の研磨製品。

13. 光ファイバコネクタフェルール端面の研磨のための上記1～8のいずれか1項記載の研磨フィルムの使用。

【 0 0 1 4 】

【作用】

本発明の研磨フィルムの研磨層は、基材上に、研磨材粒子である酸性酸化物、例えばシリカと、アミン化合物と、バインダ樹脂とを含有、分散してなる塗工液を塗布することにより形成される。塗工液の溶媒に分散されたシリカの表面は負に帯電しているため、正電荷を有するアミン化合物は電気的引力により負電荷を有するシリカ表面に吸着する。このため研磨加工において光ファイバ端面とシリカが直接接触することを防ぐことができる。またアミン化合物は石英ガラスからなるガラスファイバ表面に選択的に吸着し保護層を形成するので、ガラスファイバ部分の研磨速度が低下し、また軟質材料であるガラスファイバ表面の研磨衝撃を低減させる。さらにアミン化合物が介在することにより潤滑作用を生じ、結果として加工段差が防止され、スクラッチや加工歪みを生じることなく研磨加工されるものと考えられる。

【 0 0 1 5 】

一方、ジルコニアフェルールはアルカリ性酸化物であり、酸性酸化物とアルカリ性酸化物といった異種の無機酸化物が接触する場合、その界面で電子の授受を伴う加成反応が生じ、ジルコニア表面に複合酸化物の層が形成される。この複合酸化物の層は元のジルコニアよりも硬度が低下するので、ジルコニアの研磨除去速度が上昇して光ファイバコネクタフェルール端面での異種材料間の加工段差の発生を防止すると考えられる。さらに、アミン

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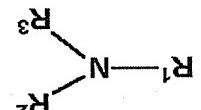
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[ 0 0 2 1 ]

其中、 $R_1$ ~ $R_{20}$ 为质量原子数 1~20 的质模量化水素基及质模量化水素基表示式、 $R^2$ 、 $R^3$ 为  
独立乙、水素原子及质模量化水素基 1~20 的质模量化水素基及质模量化水素基表示式。质  
模量化水素基乙乙乙、直链及环分枝的饱和和双键和的脂肪族量化水素基、脂肪族量化水  
素基、芳香族量化水素基及基团化水素基等分子。质模量化水素基乙乙乙、质素原子数  
1~20 的质模量化水素基、丁基与二甲基十六基等分子乙乙乙。

[ 0 0 2 0 ]



[ 5 ]

【 0 1 9 】

第七章 化合物的合成

【0018】本器皿可以用以分析稀有气体及其化合物、下配備造式(1)下表之机子1~3號

[ 8 1 0 0 ]

本器明伦用以练习研磨材料于块、酸性研化物、特以酸性金属酸化物而为口、侧无底、之  
力为、酸化锡 ( $\text{SnO}_2$ )、酸化亚砷、乙机之②槽以上之混合物亦为掌研磨机之。特  
光学于丁口之练习于研磨面上研磨之法、之力为好求之。

L4 T001

、新日鉄黒川鉱業所の新規開拓地。

从使用工具到电气控制，研磨加工时的加压等机械、玻璃在以提高玻璃中的杂质效果的研磨机的原动机、研磨机加工时的电气控制等。

以下，本說明文的範例將使用以下的工具，向您說明如何詳解說明書。

## 【堯明の美魔の形態】

[ 9 T 0 0 ]

可能比你多的機會多。

工之光效，本器明之研磨刀之以之光效，其上研磨工程之光效工之光效

此部分物块丁以力力学宗系与之共力5之化等的作用见下式，瞬时变化物飞出与之共力的表

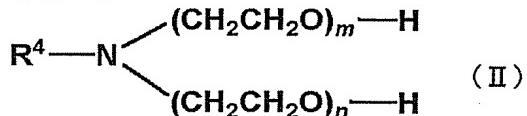
ー、mー又はpーオクチルアニリン、oー、mー又はpードデシルアニリン、oー、mー又はpーテトラデシルアニリン、oー、mー又はpーエトキシアニリン、これらの2種以上の混合物などが挙げられる。上記アルキル基、アルケニル基、アルコキシ基、アルケニルオキシ基はすべての位置異性体、光学異性体も意味するものとする。また、本発明は、これらに限定されるものではない。

## 【0022】

また本発明に用いられる他の好ましいアミン化合物としては、下記構造式(II)で表されるモノ、ジ、トリアルカノールアミン又はポリオキシエチレンアミン化合物が挙げられる。

## 【0023】

## 【化6】



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## 【0024】

式中、R<sup>4</sup>は水素原子、炭素原子数1～20の炭化水素基又は置換炭化水素基を示す。m及びnは0～8の整数であり、m+n=1～8である。炭化水素基としては、直鎖又は分岐の飽和又は不飽和の脂肪族炭化水素基、脂肪族環状炭化水素基、芳香族炭化水素基が挙げられる。置換炭化水素基の置換基としては、炭素原子数1～20のアルコキシ基、アルケニルオキシ基等が挙げられる。

## 【0025】

構造式(II)で表されるモノ、ジ、トリアルカノールアミン又はポリオキシエチレンアミン化合物の具体例としては、ヒドロキシエチレンドデシルアミン、ポリオキシエチレンドデシルアミン、ポリオキシエチレンオクタデシルアミン、ポリオキシエチレンオレイルアミン、モノエタノールアミン、ジエタノールアミン、トリエタノールアミンなどが挙げられる。本発明は、これらに限定されるものではない。

## 【0026】

これらのアミン化合物を単独若しくは二種類以上を任意の割合で添加した研磨フィルムは、高研磨面品質を達成することが可能である。

本発明に用いるこれらのアミン化合物の添加濃度は、研磨材粒子100質量部に対して0.5～20.0質量部の範囲であり、好ましくは1.0～10.0質量部である。これらの添加剤の添加量が0.5質量部より少ないと目的とする効果の発現が不十分となり、スクラッチが生じる恐れがある。また、20.0質量部を超えて添加しても効果の更なる向上は見られない。

## 【0027】

本発明の研磨フィルムの製造過程においてアミン化合物は、研磨層用の塗工液製造の任意の工程において適宜添加すればよい。例えば、バインダ樹脂を溶媒で溶解した樹脂液中に研磨材粒子とアミン化合物の必要量を添加し、混合分散すればよい。また研磨材粒子とアミン化合物とを秤量し、適量の溶媒を加えて攪拌混合し、これにバインダ樹脂を溶媒で溶解した樹脂液を加えて混合分散してもよい。これによって研磨材粒子をアミン化合物により表面処理又は湿潤処理することができる。

## 【0028】

また本発明の研磨材粒子としては、粒子径20～30nmの一次粒子の分散液を噴霧乾燥により造粒、作製される平均粒子径1～10μmの球状の造粒粒子も使用できる。この球状の造粒粒子は、一次粒子の分散液の粒子濃度、噴霧方法により造粒粒子の細孔容積や粒子径を任意に制御することができ、また、乾燥温度により一次粒子同士の結合力すなわち粒子硬度を調整できる。

本発明に使用される球状造粒粒子の平均粒子径は、好ましくは0.5～30.0μmであり、より好ましくは1.0～10.0μmである。造粒粒子の粒子径は研磨除去量に影響

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[ 0 0 2 9 ]

方大志《方文慎向游集》。

[ 0 8 0 0 ]

[ 100 ]

[ 0 0 3 2 ]

[ ε ε 0 0 ]

本說明的研磨圓盤成形方式為的墊工盤之使用要訖於工藝化削材使用之範例。變化削材之  
剖面如乙狀、半圓形等之零件一小方等皆可利用。例如螺、八半軸承子之工藝化削材之  
一 50

乙30~70質量部職員使用する効適度である。

種類以上任意の割合で使用可能。

加工費實質上是生產經營的工具或資金。

磨削作用用以去除不需要的材料或尺寸的手工器具称为刮刀。

这的大小与布道规模有关，研磨材料粒子越大越难，能来的微粒子越大则研磨时间少且效率高。研磨一定量的研磨材料时，研磨时间与研磨材料粒子大小与效率成反比，研磨时间与研磨材料的密度成正比。

卷之三

本證明証明該用之批子造數子以未築成之主用之於此。900°C以下、好率之<br/>420°C以下、1~5時間、酸素、空氣、不活性為之在已需要圓錐下已築成之電  
力之使用才可之乙也乙者。燒成溫度為900°C之時之圓錐下之結合力才為  
之數子燒度為大者<好>、結果之乙一次數子乙好<造數子好>圓之研磨材料數子之乙  
作用才之乙加5異種磨材料之研磨圓之研磨量著化上之生乙多加工段差為大者<好之傾向  
好也、420°C之時之圓錐表面之數子表面之好>一此基於水縮合之乙消失  
與之乙之乙好（參考文獻乙之乙好）。

首先本器皿以使用之机子造粒料之制孔率填进0.1~1.0cc/每立方厘米。0.1cc/每立方厘米第一次粒子为球状或颗粒状，造粒料之硬度为大者<0.1cc/每立方厘米。0.1cc/每立方厘米的研磨量差比0.1cc/每立方厘米加工段差为大者<0.1cc/每立方厘米。1.0cc/每立方厘米第一次粒子为球状或颗粒状，造粒料之硬度为大者<0.1cc/每立方厘米加工段差为大者<0.1cc/每立方厘米。

0.5 μm 未満では、研磨力が他の方法よりも、30倍以上の加工能率をもたらす。

ト、トリレンジイソシアネート、キシリレンジイソシアネート、イソホロンジイソシアネート、ナフチレンジイソシアネートなどが挙げられる。これらの硬化剤は単独もしくは二種類以上任意の割合で使用される。

#### 【0034】

本発明の研磨層を形成するための塗工液に使用される溶媒は、特に限定されないが、例えば、アセトン、メチルエチルケトン、メチルイソブチルケトン、シクロヘキサン、イソホロン、酢酸エチル、酢酸ブチル、テトラヒドロフラン、トルエン、キシレン、エタノール、イソプロパノールなどが挙げられる。これらの有機溶媒は、バインダ樹脂、硬化剤を均一に溶解でき、さらに、研磨材粒子が安定に分散され、塗布後、完全に乾燥除去されるように単独もしくは二種類以上任意の割合で使用される。10

また本発明の研磨層を形成するための塗工液には必要に応じて他の界面活性剤やカップリング剤などを使用することが出来る。

#### 【0035】

本発明の研磨フィルムは、バインダ樹脂を溶媒で溶解した樹脂液中に研磨材粒子とアミン化合物の必要量を秤量、混合し、サンドミルなどの分散機で、研磨材粒子を樹脂液中に均一に分散し、基材上へ塗布することにより製造される。塗工液中の固形分の割合は、好ましくは30～70質量%である。基材上への塗布手段としては、例えば、ワイヤーバーコーター、グラビアコーティング、リバースコーティング、ナイフコーティング、ノズル、ダイコーティングなどが使用できる。この様な塗布手段により、基材上に塗工液を乾燥質量で5～20g/m<sup>2</sup>程度となるように塗布し、40～120℃で0.5～72時間乾燥又は硬化処理する20ことにより、基材へ研磨層が固着される。20

#### 【0036】

##### 【実施例】

以下、本発明を実施例によりさらに具体的に説明する。

##### 実施例1、比較例1及び2

メチルエチルケトン(44質量部)に、バインダ樹脂としてポリエステルウレタン系樹脂UR1350(東洋紡績株式会社製：固形分33質量%) (35質量部)を溶解し樹脂液を作製した。これに研磨材粒子としてヒュームドシリカ(粉碎シリカ)(FQ3000：株式会社フジミインコーゴーポレーテッド製、平均粒径3～4μm) (20質量部)、添加剤として1級アミン(化合物名POE(7)ドデシルアミン)(ナイミーンL-207：日本油脂株式会社製) (1質量部)を加え、サンドミルで均一に分散した後、濾過精度75μmのフィルタリングを施し塗工液を作製した。30

#### 【0037】

比較例1として実施例1において添加剤としての1級アミンを加えずに、バインダ樹脂の量を36質量部としたもの、比較例2として平均粒子サイズが約50nmの微粒子シリカからなる研磨材粒子(オルガノシリカゾルM E K-S T (30質量%)) (80質量部)(溶媒メチルエチルケトン)とバインダ樹脂としてシリコーン樹脂(KR-9218：信越化学工業株式会社製) (20質量部)を混合攪拌処理して塗工液を作製した。

作製した塗工液をナイフコーティングにより、易接着処理(ポリエステル系樹脂を約0.1μm程度塗布したもの)を施した厚み75μmのポリエチレンテレフタレートフィルム(S40G-2：帝人株式会社製)上に塗布した。コーティングの乾燥炉温度は120℃に設定し、塗布後、24時間以上室温放置した。乾燥後の塗布厚みは5μmとした。

#### 【0038】

##### 実施例2～8、比較例3～8

次に、研磨材粒子として球状造粒粒子を用いた実施例について述べる。

有機溶媒としてメチルエチルケトン(44質量部)、バインダ樹脂としてポリエステルウレタン系樹脂UR1350(東洋紡績株式会社製：33質量%) (35質量部)を用いて樹脂液を作製した。これに研磨材粒子としてシリカ造粒研磨材粒子P500(触媒化成工業株式会社製) (20質量部)と、表1に示す添加剤(1質量部)を混合攪拌し、サンドミルで均一に分散した後、濾過精度75μmのフィルタリングを施し塗工液を作製した。50

510. 2) 电子显微镜的分辨率(Axio Plan、观察倍率×  
510. 2) 电子显微镜的分辨率(Axio Plan、观察倍率×  
AFM、TOMO Metric、EDX、IORE2000、DIORE100  
SEM)比干涉仪二极管光路的测量灵敏度高。加工段差时原子弹力显微镜(

40 美麗開拓者以此較弱的研磨力而比之為少，適當的研磨工程以能化光刀而減少刀刃之  
[0041] 41 美麗開拓者以此較弱的研磨力而比之為少，適當的研磨工程以能化光刀而減少刀刃之

〔0041〕

【卷一】

三为小儿之搽拭会社敷）、比较例7、8途中性化合物（花王株式会社製）也表示。

美輪例2、3付1級73頁、美輪例4、5付3級73頁、美輪例6～8付1牛川73頁  
化工具1付、計算機73頁。比輪例3付添加削除添加73頁。未だ実施例付有付73  
化合物(日光化会社日本油脂株式会社製)乙丸多丸、比輪例4～6付酸性化合物(日光分

【 6 8 0 0 】

反射減衰量の値は、

$$RL = 10 \log_{10} (P_r / P_i) [dB]$$

によって算出される。ここで、 $P_i$ は入力した光パワー、 $P_r$ は反射した光パワーをそれぞれ示す。反射減衰量の値が $-10\text{ dB}$ と仮定すると、光ファイバ端面に $500\mu\text{W}$ の光が入射すれば、 $50\mu\text{W}$ の光が光源に向かって反射することになる。つまり、 $10\%$ のエネルギーが損失することになる。結果を表2に示す。

【 0 0 4 2 】

【表2】

	スクラッチ	加工段差	樹脂付着	反射減衰量
実施例1	ややあり	-29 nm	なし	59 dB
比較例2	なし	+10 nm	あり	57 dB
比較例3	あり	-81 nm	なし	42 dB
実施例2	なし	-2 nm	なし	62 dB
実施例3	なし	-5 nm	なし	60 dB
実施例4	なし	-20 nm	なし	60 dB
実施例5	なし	-18 nm	なし	60 dB
実施例6	なし	-5 nm	なし	66 dB
実施例7	なし	-8 nm	なし	63 dB
実施例8	なし	-13 nm	なし	62 dB
比較例3	あり	-110 nm	なし	58 dB
比較例4	あり	-153 nm	なし	52 dB
比較例5	あり	-162 nm	なし	55 dB
比較例6	あり	-109 nm	なし	53 dB
比較例7	あり	-89 nm	なし	55 dB
比較例8	あり	-81 nm	なし	53 dB

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【 0 0 4 3 】

評価した結果、実施例1に対しアミン化合物を添加しなかった比較例1では、ガラスファイバ及びジルコニアフェルールのキズも多発し、反射減衰量も低かった。平均粒子サイズが約 $50\text{ nm}$ の微粒子シリカからなる研磨材粒子とシリコーン樹脂を用いて比較例2は樹脂付着が発生した。実施例1のアミン化合物を添加した研磨フィルムでは、ガラスファイバに若干のキズは残るものジルコニアフェルールはキズもなく、反射減衰量も比較例1より高くなつたことから、アミン化合物の効果によって研磨品質および光学特性を向上させることが可能であることが確認された。

【 0 0 4 4 】

球状造粒粒子を研磨材粒子として用いた実施例2～8の研磨フィルムは、ガラスファイバ

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。また、反射減衰量は常に  $60 \text{ dB}$  以上であり、光信号の減衰が非常に少ない精密測定が可能である。  
【実験の結果】  
以上のように本実験の結果は以下に要約される。  
(1) 45 [ ]  
反射率は比較的  $3 \sim 8$  の範囲で  $\pm 1\%$  程度、反射損失は  $10$  デシベル以内で  $\pm 1\%$  程度である。  
また、反射減衰量は大体  $< 55 \text{ dB}$  である。  
【実験の結果】  
以上のように手が無い、加工精度が  $20 \text{ nm}$  以下であり、反射損失は  $5 \sim 10 \text{ dB}$  である。  
【実験の結果】  
以上のように手が無い、反射減衰量が  $60 \text{ dB}$  以上であり、光信号の減衰が非常に少ない精密測定が可能である。

【證明の効果】

[ 0 0 4 5 ]

部分的火力大于对手，加工质量也有20nm以下的优势，油脂封装电阻为5Ω左右。发光二极管的发光强度比对手高出60dB以上，光信号的衰减也非常小。在竞争对手面前，我们有绝对的优势。

フロントページの続き

(51) Int.Cl.<sup>7</sup>

F I

テーマコード (参考)

B 2 4 B 19/00 6 0 3 Z  
B 2 4 B 21/00 Z

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F ターム(参考) 3C049 AA05 AA09 CB02

3C058 AA05 AA09 CB02

3C063 AA03 AB07 BB01 BB07 BB08 BB15 BC03 BG08 EE01

